



USDA Foreign Agricultural Service

# GAIN Report

Global Agriculture Information Network

Template Version 2.09

Voluntary Report - public distribution

**Date:** 1/17/2006

**GAIN Report Number:** CH6001

## China, Peoples Republic of

### FAIRS Product Specific

### Fresh and Frozen Poultry Product Standard

### 2006

**Approved by:**

Casey Bean

U.S. Embassy Beijing, Office of Agricultural Affairs

**Prepared by:**

Wu Bugang

---

**Report Highlights:**

This is an UNOFFICIAL translation of the People's Republic of China National Standard on Fresh and Frozen Poultry Products (GB16869-2005) and should be used as a guide only. Exporters should carefully discuss the regulation and its application with Chinese importers to ensure their interpretation is accurate. According to the new standard, a zero tolerance for Salmonella sp. and hemorrhagic Escherichia coli (O157: H7) still applies on fresh/frozen poultry products, but there is no requirement for testing of Listeria sp.

---

Includes PSD Changes: No

Includes Trade Matrix: No

Unscheduled Report

Beijing [CH1]

[CH]

**Summary**

This standard details the technical requirements for testing, hygiene, labeling, packaging, and storage requirements for fresh and frozen poultry products. It replaces the previous poultry standard (GB16869-2000) (CH1060) and is the final version of the draft standard on poultry products (CH3052). Compared with the poultry standard from 2000, the differences include: 1) revising the coliform bacteria limit for frozen poultry products to  $5 \times 10^3$  MPN or less per 100g, 2) testing for lactic Escherichia coli changed to hemorrhagic Escherichia coli (O157: H7), 3) a zero tolerance standard for salmonella and hemorrhagic escherichia coli (O157: H7) still applies, but there is no requirement for testing of Listeria sp. In table 3, Chinese officials explained to FAS Beijing that "0/25g" means zero tolerance in a 25 gram sample for Salmonella or E.coli 0157.

The Standardization Administration of China (SAC) and General Administration for Quality Supervision Inspection and Quarantine (AQSIQ) issued this standard on March 23, 2005, but the standard went into effect on January 1, 2006.

BEGIN TRANSLATION

**Fresh and Frozen Poultry Products (GB16869-2005)****Preface**

Chapter 6 of this standard is recommended, while the others are compulsory.

This standard will replace GB16869-2000 *Fresh and frozen poultry products*.

This standard has the following changes from GB16869 *Fresh and frozen poultry products*:

1. Limits are no longer specified for methamidophos and clenbuterol.
2. A method for calculating the examining gores and hard feathers, not counting areas of  $0.5\text{cm}^2$  or less.
3. Certain technical requirements are changed.
4. The core temperature for frozen poultry products is changed to  $-18^\circ\text{C}$  or lower.
5. The water ratio for thawed poultry is changed to 6% or less.
6. The plumbum limit is changed to 0.2mg/kg or less.
7. The BHC residue limit is changed to 0.1mg/kg (for the whole sample) and 1mg/kg (for fat).
8. The limit for coliform bacteria for frozen poultry products is changed to  $5 \times 10^3$  MPN or less per 100g.
9. The salmonella limit is changed to "0/25 g".
10. The lactic escherichia coli limit is changed to the limit of hemorrhagic escherichia coli (O157: H7) , 0/25 g.
11. The method of determining stilboestrol is changed to that in regulation SN0672.

The sampling method and number of permissible defects described in the routine examination, and acceptance examination in Chapter 6 of this standard are same as in the Examination Standard I and Examination Standard II of the *Sampling Methods of Pre-packed Food*, CAC/RM 42-1969.

Appendix A of this standard is a normative appendix.

This standard is drafted by the China National Food Industry Standardization Technology Committee, the Food Hygiene Standardization Committee of the Health Standardization Technology Committee of the Public Health Ministry of the People's Republic of China.

This standard falls under the China National Food Industry Standardization Technology Committee.

The units which drafted this standard are the Health Supervision and Examination Institute of Public Health Ministry, Secretary Department of China National Food Industry Standardization Technology Committee, Health Supervision Institute of Shanghai Health Administration Bureau as well as the Butchering Technology Appraisal Center of China Domestic Trade Bureau, the Quality Examination Center of Livestock & Poultry Products of the Agriculture Ministry, China Meat Association, Beijing Entry-Exit Inspection and Quarantine Administration Bureau and the Shenzhen Entry-Exit Inspection and Quarantine Administration Bureau of the People's Republic of China.

The main drafters of this standard are Hao Yu, Han Yulian, Gu Jingyu, Ran Binqi, Lin Linan, Yang Xiaoming, Liu Hong, Liu Suying, Li Chunfeng and Tan Ying.

The units which drafted Appendix A are the Nutrition & Food Health Research Institute of China Preventive Medicine Science Academy, and the Food Health Supervision Institute of the Public Health Ministry of the People's Republic of China.

The main drafters of Appendix A are Chen Huijing, Wang Xuqing, Yang Dajin and Wu Guohua.

This standard will replace its predecessors: GB 2710-1996, GB 16869-1997 and GB 16869-2000.

## Fresh and frozen poultry products

### 1. Applicable Scope

This standard expressly describes the technical specifications, examination methods, examination rules and regulations, symbols, and packaging and storage of fresh and frozen poultry products.

It applies to not only fresh and frozen poultry products made of live and healthy poultry by butchering, processing and packing, but also those which are unpackaged.

### 2. Referential Standards

Some clauses in the following standards become a part of this standard through citation. Revisions to dated standards will not become a part of this standard (excluding corrected errors), although parties making agreements based on this standard are encouraged to consider using the latest edition of these standards. For undated standards, the latest editions will apply to this standard.

GB 191	<i>Figured label of packaging, storage and transportation,</i>
GB/T 4879.2-2003	<i>Microbiological Examination of Food Hygiene—Aerobic Bacterial Count</i>
GB/T 4879.3-2003	<i>Microbiological Examination of Food Hygiene—Detection of Coliform Bacteria</i>
GB/T 4879.4-2003	<i>Microbiological Examination of Food Hygiene—Examination of Salmonella</i>
GB/T5009.11-2003	<i>Maximum level of arsenic and inorganic arsenic in food</i>
GB/T5009.12-2003	<i>Maximum level of plumbum in food</i>
GB/T5009.17-2003	<i>Maximum level of hydrargyrum and organic hydrargyrum in food</i>
GB/T5009.19-2003	<i>Maximum Residue limit of BHC and DDT in food</i>
GB/T5009.44-2003	<i>Analysis of Meat and Meat Product Health Standard</i>
GB/T6388	<i>Delivery &amp; receiving labels for transportation and package</i>
GB 7718	<i>General standard of food labeling</i>
GB/T14931.1-1994	<i>Determination of acheomycin, aureomycin, terramycin residues in livestock &amp; poultry meat (high performance liquid chromatography)</i>
SN 0208-1993	<i>Determination of 10 sulfanilamide residues in export meat</i>
SN/T 0212.3-1993	<i>Determination of Clopidol residues in export meat, Propionyl gas chromatography</i>
SN 0672-1997	<i>Determination of stilboestrol residues in export meat and meat product Radioimmunity</i>
SN/T 0973-2000	<i>Determination of hemorrhagic coliform bacteria 0157: H7 in export meat and meat product</i>

### 3. Glossary and definition

The following glossary and definitions apply to this standard.

#### 3.1 Fresh poultry products

Poultry products made of live poultry after butchering, processing and pre-cooling, including whole poultry, cut meat (poultry meat, wings, legs, etc.) and other parts (heads, necks, bowels, feet, etc.).

3.2 Frozen poultry product

Poultry products that are made of the live poultry after butchering, processing and freezing, including whole disemboweled poultry, cut meat (poultry meat, wings, legs, etc.) and other parts (heads, necks, bowels, feet, etc.).

3.3 Impurities

Matter ordinarily seen as a waste or polluted substance such as yellow skin, feces, bile, and other undesired substances (plastic, metal, remaining meal, etc.)

**4. Technical Requirements**

4.1 Raw materials

Live poultry from non-epidemic stricken regions which have passed related quarantines and examinations before butchering.

4.2 Processing

After butchering, poultry will not be processed until it has passed related quarantines and examinations, then processing may proceed.

4.2.1 Finishing

Every external wound, blood spot or blood pollution, feather ends and other some such will be removed or cut away from the poultry.

4.2.2 Cutting

Poultry can only be cut after pre-cooling, and it will go through bleeding, packaging and freezing storage in 2 hours or less.

4.3 Freezing

The frozen products will be stored at a core temperature of -18? or lower in 12 hours.

4.4 Sensory properties

Refer to Table 1 for details.

Table 1

Item	Fresh product	Frozen product (unfrozen)
Tissue property	Elastic muscle, resuming its normal form after being pressed upon by a finger	Muscle is slow or hard to resume its original form after being pressed upon by a finger
Color	Surface skin and muscle are shining, i.e., full of poultry shine	
Smell	A poultry-only smell, with no strange smell	
Gravy	Clear with some fat in the soup and tasting only of poultry.	
Gore (counted in gore area/S)/cm <sup>2</sup> S>1 0.5< S=1 S=0.5	Not passed Cannot exceed 2% of the sample Immaterial	

Hard feathers (feathers of 12mm or longer or feather ends of 2mm or longer)/(piece/10kg) =	1
Impurities	Not found
Note: The gore area means a blood spot found on a whole bird or in cut meat.	

## 4.5 Physical and chemical index

For the details on fresh and frozen poultry products, refer to Table 2.

Table 2

Item		Index
Water from thawed poultry product/(%)=		6
Volatile electropositive ammonia/(mg/100g) =		15
Hydrargyrum (Hg)/(mg/kg) =		0.05
Plumbum (Pb) /(mg/kg) =		0.2
Arsenic (As) /(mg/kg) =		0.5
BHC/(mg/kg)	Counted in a whole sample when fat is lower than 10%, =	0.1
	Counted in fat when fat is higher than 10%, =	1
Chlorophenothane/(mg/kg)	Counted in a whole sample when fat is lower than 10%, =	0.2
	Counted in fat when fat is higher than 10%, =	2
Atgard/(mg/kg) =		0.05
Acheomycin/(mg/kg)	Muscle =	0.25
	Liver =	0.3
	Kidney =	0.6
Aureomycin/(mg/kg) =		1
Terramycin/(mg/kg)	Muscle =	0.1
	Liver =	0.3
	Kidney =	0.6
Sulfamethazine/(mg/kg) =		0.1
Clopidol/(mg/kg) =		0.01
Stilboestrol/(mg/kg) =		Not found

## 4.6 Microbe index

See Table 3.

Table 3.

Item	Index	
	Fresh products	Frozen products
Aerobic Bacterial Count/(cfu/g) =	$1 \times 10^6$	$5 \times 10^5$
Coliform Bacteria/(MPN/100g) =	$1 \times 10^4$	$5 \times 10^3$
Salmonella	0/25g <sup>a</sup>	
Hemorrhagic escherichia coli (O157:H7)	0/25g <sup>a</sup>	
a: To take 5 samples.		

## 5. Examination methods

### 5.1 Sensory properties

Examination can be done only after the samples are thawed.

#### 5.1.1 Tissue property, color and smell

Put the samples in natural light or in a sensory examination room with a natural light after the microbe examination. Check them by sensory contact. Determine their colors and smells by your eyes and nose respectively, for instance.

#### 5.1.2 Gravy after heating

Cut the sample (in accordance with Clause 6.5.4), put it in a flask and add 100ml of water, and heat it to 50-60°C after covering the flask. After this smell it with the nose and check the gravy and fat after boiling. When cooled to room temperature, you can taste the gravy.

#### 5.1.3 Gore

After examining tissue properties, colors and smell, you can determine the gore in the following method.

In a basic vessel, compute the ratio between the gores of  $0.5\text{cm}^2 < S = 1\text{ cm}^2$  and the total product quantity in the same vessel in Formula (1):

$$X = \frac{A_1}{A} \times 100 \dots \dots \dots (1)$$

Where:

X—the ratio between the gores of  $0.5\text{ cm}^2 < S = 1\text{ cm}^2$  and the total product quantity in a basic vessel (counted as whole birds, or cut meat, or as legs or wings), %,

A— the total product quantity in a basic vessel, and

$A_1$ —the gores of  $0.5\text{cm}^2 < S = 1\text{ cm}^2$  in a basic vessel.

#### 5.1.4 Hard feathers

Do this determination while checking the tissue property, color and smell of a sample. For instance, you can check the hard feathers with a vernier caliper at the precision of 0.05mm and calculate them for every 10kg in a basic vessel in accordance with Formula (2):

$$X_1 = \frac{A_2}{m} \times 100 \dots \dots \dots (2)$$

Where:

$X_1$ —The quantity of hard feathers in every 10 kg in a basic vessel,

$A_2$ —The actual quantity of hard feathers in a basic vessel, and

$m$ —The actual weight of a basic vessel, in kg.

#### 5.1.5 Impurities

Determine this while checking the tissue property, color and smell of a sample.

### 5.2 Unfrozen water

#### 5.2.1 Instruments and tools

Electronic scale: to a precision of 1g,

Thermometer: -10°C~50°C, at a scale of 0.5°C, and

A porcelain plate and an iron screen.

#### 5.2.2 Determination program

Put a piece of iron screen on the porcelain plate. Keep the screen 2cm over the porcelain plate. Take 1,000g~2,000g from the sample selected (in accordance with Clause 6.5.2) and put it on the iron screen after weighing. Cover it with a piece of plastic film and let it thaw naturally at a temperature of 15°C~25°C. When it reaches a temperature of 2°C~3°C, take the film away and weigh it on the electronic scale. After that, you can put it back on the iron screen and weigh it after 30 minutes. Repeat the above-mentioned operations until there is a difference of 2.0g or less after 2 operations.

#### 5.2.3 How to describe a determined result

Work out the thawed water ratio in accordance with Formula (3):

$$X_2 = \frac{m-m_1}{m} \times 100 \dots \dots \dots (3)$$

Where:

$X_2$ —The thawed water ratio of a sample,

$m$ —The weight of a sample before being thawed, in gram (g) and

$m_1$ —The weight of a sample after being thawed, in gram (g).

You can keep an integral for a final result.

#### 5.3 Volatile electropositive ammonia

Determine it in accordance with Clause 4.1 of GB/T 5009.44-2003.

#### 5.4 Hydrargyrum

Determine it in accordance with GB/T 5009.17-2003.

#### 5.5 Arsenic

Determine it in accordance with GB/T 5009.11-2003.

#### 5.6 Plumbum

Determine it in accordance with GB/T 5009.12-2003.

#### 5.7 BHC and Chlorophenothane

Determine it in accordance with GB/T 5009.19-2003.

#### 5.8 Atgard

Determine it in accordance with the examination method shown in Appendix A.

#### 5.9 Acheomycin, Aureomycin and Terramycin

Determine it in accordance with GB/T 14931.1-1994.

#### 5.10 Sulfamethazine

Determine it in accordance with SN 0208-1993.

#### 5.11 Clopidol

Determine it in accordance with SN/T 0212.3-1993.

#### 5.12 Stilboestrol

Determine it in accordance with SN 0672-1997.



### 5.13 Aerobic Bacterial Count

Determine it in accordance with GB/T 4789.2-2003.

### 5.14 Coliform Bacteria

Determine it in accordance with GB/T 4789.3-2003.

### 5.15 Salmonella

Determine it in accordance with GB/T 4789.4-2003.

### 5.16 Hemorrhagic escherichia coli 0157: H7

Determine it in accordance with SN/T 0973-2000.

### 5.17 Temperature of product core

#### 5.17.1 Thermometer

Non-mercury glass thermometer or other temperature instrument at a scale of -20°C~50°C.

#### 5.17.2 Program

Drill the meat sample with a drilling bit a little larger in diameter than the thermometer and then push the thermometer (or other temperature measuring tool) into the meat as soon as the drill bit is removed from the sample. After the reading stabilizes, read it from the thermometer.

## 6. Examination regulations

### 6.1.1 Routine examination

6.1.1.1 Do a routine examination whenever any of the following conditions are found:

- a) Only a batch of product,
- b) A change in the supply region of live poultry,
- c) The 1<sup>st</sup> batch of product from a new plant,
- d) 6 production months without a stop or reproduction after a period of rest,
- e) A considerable change is found between this examination result and the last routine examination, and
- f) A demand is required by local quality supervision or health inspection departments.

6.1.1.2 The routine examination consists of the items given in Tables 1, 2 and 3.

### 6.1.2 Acceptance examination

6.1.2.1 All products can be delivered only after the acceptance examination.

6.1.2.2 The acceptance examination consists of the items shown in Table 1, including the thawed water ratio of frozen poultry, volatile electropositive ammonia, bacterial count and coliform bacteria.

## 6.2 Batch Types

### 6.2.1 Continuous batch

A continuous batch means that the products are processed under the same conditions, the same parts of a kind of bird (including a whole bird and its meat, wings, legs, head, feet, bowels) finished and packed in the same way and delivered only once, counted in a basic packaging vessel (hereafter basic vessel).

### 6.2.2 Independent batch

An independent batch means that the products are made from the same parts of a kind of bird (including whole bird and its meat, wings, legs, head, feet, bowels), packed in the same way and delivered only once, counted in a basic vessel.

### 6.3 Sampling

#### 6.3.1 A sample of routine examination

In accordance with the batch types, you can take a sample in light of Table 4.

Table 4

Qty. of a batch (basic vessel)	Sample qty. (basic vessel)	Allowable defects (basic vessel)
600 or less	13	2
601~2,000	21	3
2,001~ 7,200	29	4
7,201~ 15,000	48	6
15,001~ 24,000	84	9
24,001~ 42,000	126	13
More than 42,000	200	19

#### 6.3.2 A sample of acceptance examination

In accordance with the batch types, you can take a sample in light of Table 5.

Table 5

Qty. of a batch (basic vessel)	Sample qty. (basic vessel)	Allowable defects (basic vessel)
600 or less	6	1
601~2,000	13	2
2,001~ 7,200	21	3
7,201~ 15,000	29	4
15,001~ 24,000	48	6
24,001~ 42,000	84	9
More than 42,000	126	13

### 6.4 Sampling and examination programs

For the sampling and examination program for fresh and frozen poultry products, refer to Figure 1.

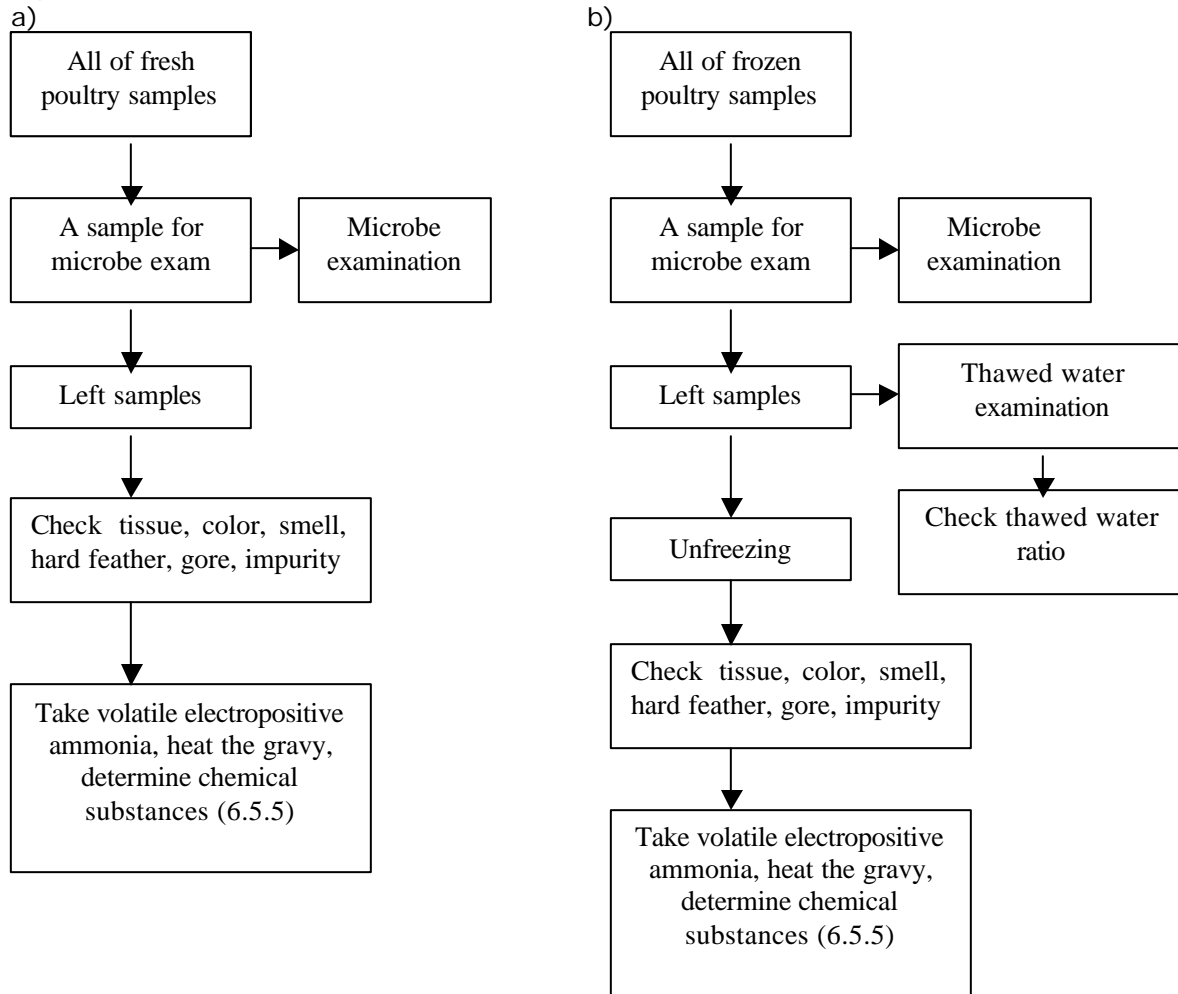


Figure 1. Sampling and examination programs for fresh and frozen poultry products

#### 6.5 How to take a sample

The following samples cannot contain any gore, hard feathers or impurities.

##### 6.5.1 Microbe examination sample

Freely select (3~5) basic vessels from all samples, take a sample of 100g from each vessel in a sterile working environment and mix them together.

Note: You can take 5 samples (25g/piece) from mixed ones for the salmonella examination and take another 5 samples (25g/piece) for the escherichia coli examination.

##### 6.5.2 Thawed water examination sample

Freely select (3~5) basic vessels from the frozen poultry samples, take 500g from every vessel, put them in the insulation tank after mixing.

##### 6.5.3 Volatile electropositive ammonia examination sample

Freely select 3 basic vessels from all the samples, take 100g from every vessel but with no fat or bone, mixing them together.

##### 6.5.4 Heated gravy examination sample

Freely select 3 basic vessels from all the samples of whole bird and its meat, wing or leg, take 100g from each type of meat, mixing them together.

##### 6.5.5 Chemical substance examination sample

Freely select 3 basic vessels from all the samples, take about 200g from the edible part, mixing them together.

#### 6.6 Determination standard and reexamination

##### 6.6.1 Types of defect

6.6.1.1 General defects: gore, hard feather, not given in a standard.

6.6.1.2 Serious defects: some items, such as poor tissue property, color, smell and heated gravy, or shown in Tables 2 and 3, or the impurity clearly seen.

##### 6.6.2 Determination of all examination results

6.6.2.1 How to determine gore and hard feather: A basic vessel is taken as a unit in the this examination.

Example 1:

All of the samples are put in 6 basic vessels and respectively in a serial number.

Final result: It fails to meet the standard because of gore in Vessel 1 and poor hard feathers in Vessel 3.

Determination: A general defect found in 2 basic vessels.

Example 2:

All of the samples are put in 13 basic vessels and respectively in a serial number.

Final result: It fails to meet the standard because of gore in Vessels 1~13 and poor hard feathers in Vessel 8.

Determination: A general defect found in 13 basic vessels.

6.6.2.2 How to determine the tissue property, color, smell, heated gravy and the examination items given in Tables 2 and 3: whenever an item is below the index of this

standard, the samples can all be seen in a serious defect.

#### 6.6.3 Routine examination determination and reexamination

6.6.3.1 When and if all the items are passed in the routine examination (Clause 6.6.1.2), this batch of products is acceptable.

6.6.3.2 When a serious defect (Clause 6.6.1.2) is found in the routine examination according to this standard, this batch of products is not acceptable and no reexamination is approved.

6.6.3.3 When a general defect (Clause 6.6.1.1) is found in the routine examination, but the amount is not greater than the figure in the allowable index shown in Table 4, this batch of products is acceptable. Whenever it is higher than the said allowable index, you will have to take the samples once again and re-examine them in accordance with Table 4 (allowable defects), and then, determine whether or not to accept this batch of products.

#### 6.6.4 Acceptance examination determination and reexamination

6.6.4.1 When and if all the items are passed in the acceptance examination (Clause 6.1.2.2), this batch of products is acceptable.

6.6.4.2 When a serious defect (Clause 6.6.1.2) is found in the acceptance examination according to this standard, this batch of products is not acceptable and no reexamination is approved.

6.6.4.3 When a general defect (Clause 6.6.1.1) is found in the acceptance examination, but not higher than the allowable index shown in Table 5, this batch of products is acceptable. Whenever the number of defects is higher than the said allowable index, you will have to take the samples once again and re-examine them in accordance with Table 4 (allowable defects), and then, determine whether or not to accept this batch of products.

### 7. Label, indication, package and storage

#### 7.1. Label and indication

##### 7.1.1 Label

The labels to be used directly for the consumers are made in accordance with GB 7718.

##### 7.1.2 Indication of transportation and storage

The indications for both transportation and storage are made in accordance with GB/T 191 and GB/T 6388.

#### 7.2 Package

The fresh or frozen poultry products will be packed with the brand new packing materials according to the relative health standards.

#### 7.3 Storage

The frozen poultry products will be stored in cold storage at a temperature of  $-18^{\circ}\text{C}$  and with a change of  $1^{\circ}\text{C}$  or less around the clock.

## Appendix A

**(Standard Appendix)**  
**Determination of Residue Limits**  
**of Organic Phosphorus Pesticide Polycomponent Left in Animal Food**

This appendix applies to the determination of residue limits of organic phosphorus pesticide polycomponent (methamidophos, dichlorvos, acephate, monocrotophos, dimethoate, disulfaton, parathion-methyl, fenitrothion, pirimiphos methyl, malathion, fenthion, parathion and ethion) left in livestock and poultry meats, milk and its products, egg and its products.

The minimum limits ( $\mu\text{g}/\text{kg}$ ) of the above-mentioned pesticides are respectively: methamidophos: 5.7, dichlorvos: 3.5, acephate: 10.0, monocrotophos: 12.0, dimethoate: 2.6, disulfaton: 1.2, parathion-methyl: 2.6, fenitrothion: 2.9, pirimiphos methyl: 2.5, malathion: 2.8, fenthion: 2.1, parathion: 2.6, and ethion: 1.7.

#### A.1 Main method

After the sample is purified, concentrated, separated (by the capillary tube vapor-phase chromatography), check it with the flame-photometric detector, determine its chemical composition and fix its quantity with the method in the external standard.

Sequence: ethamidophos, dichlorvos, acephate, monocrotophos, dimethoate, disulfaton, parathion-methyl, fenitrothion, pirimiphos methyl, malathion, fenthion, parathion and ethion.

#### A.2 Reagent

Unless otherwise specified, the reagents in this experiment are all pure analytical reagents. The water to be used in the experiment is prepared in accordance with the Grade II water in GB/T 6682.

A. 2.1 Acetone: re-distillation.

A.2.2 Methylene chloride: re-distillation.

A.2.3 Ethyl acetate: re-distillation.

A.2.4 Hexamethylene: re-distillation.

A.2.5 Sodium chloride

A.2.6 Anhydrous sodium sulfate

A.2.7 Gel: Bio-Beads S-X3 (or equivalent to the gel of Bio-Beads S-X3), 200~400 meshes.

A.2.8 Standard organic phosphorous pesticides: ethamidophos, dichlorvos, acephate, monocrotophos, dimethoate, disulfaton, parathion-methyl, fenitrothion, pirimiphos methyl, malathion, fenthion, parathion and ethion, at a purity of 99% or higher.

A.2.9 How to prepare the standard solution of organic phosphorous pesticide

A.2.9.1 To prepare the standard storage liquid of monomeric organic phosphorous pesticide: weigh the products of organic phosphorous pesticides, 0.0100g for each type, pour them into 25ml flasks, and dissolve them respectively with ethyl acetate (at a concentration of 400 $\mu\text{g}/\text{ml}$ ).

A.2.9.2 To mix the standard liquid of organic phosphorous pesticide: Before a determination, take the standard storage liquids of monomeric organic phosphorous pesticide (A.2.9.1) and pour them into the 10ml flasks, blow it with nitrogen gas, dilute and

fix them with fresh milk taken and purified according to Clause A.5.1.3 and Clause A.5.2. In the mixed standard liquid, the concentrations ( $\mu\text{g/mL}$ ) of the above-mentioned organic phosphorous pesticides are methamidophos: 16, dichlorvos: 80, acephate: 24, monocrotophos: 80, dimethoate: 16, disulfaton: 24, parathion-methyl: 16, fenitrothion: 16, pirimiphos methyl: 16, malathion: 16, fenthion: 24, parathion: 16, and ethion: 8.

Note: It is necessary to prepare the standard storage and utilization liquids of dichlorvos to determine if it is only dichlorvos.

### A.3 Instrument

A.3.1 Gas chromatography: with a flame-photometric detector and capillary tube vapor-phase chromatography.

### A.3.2 Rotatory evaporator

A.3.3 Gel purifying column: it is 30cm long and 2.5cm in diameter and it has a glass piston post and some glass fiber at the bottom. Put the gel soaked in the ethyl acetate and hexamethylene (1:1) into the column and keep it in the eluting agent because the column bed is 26cm high.

### A.4 How to prepare a sample

A.4.1 Egg and its product: break the egg and beat the contents.

A.4.2 Meat and its product: cut it into small parts after removing any tendon or bone and make it to meat gruel.

A.4.3 Milk and its products: mix it.

### A. 5 Analysis program

#### A.5.1 Taking, distributing and concentration

A.5.1.1 Egg and its products: take a sample of 20g (at a precision of 0.01g) and put it into a flask of 100mL, add 5ml of water (to add it according to the water content of the sample and keep a total water content of 20g, often add 5ml of water because a fresh egg contains 75% water) and with 40ml of acetone, and shake it for 30 minutes. Add 6g of sodium chloride and shake it, add 30ml of methylene chloride and shake it for 30 minutes. Take 35ml of cleaning liquid and put it into the rotatory evaporating glass through anhydrous sodium sulfate, concentrate it to about 1mL, add the ethyl acetate and hexamethylene (1:1) and concentrate it once again. After that, you can repeat the said operations for 3 times and concentrate it to about 1ml.

A.5.1.2 Meat and its products: take a sample of 20g (at a precision of 0.01g) and put it into a 100ml flask, add 6ml of water (add according to the water content of the sample and keep it at a total water content of 20g, often add 6ml because fresh meat contains 75% water). After that, follow the procedure in Clause A.5.1.1.

A.5.1.3 Milk and its product: take a sample of 20g (at a precision of 0.01g) and put it into a 100ml flask, add 6ml of water (unnecessary to add water, so prepare it with only acetone). After that, you can follow the procedure in Clause A.5.1.1.

#### A.5.2 Purification

Wash the concentrated solution (prepared in Clause A.5.1) with ethyl acetate and hexamethylene (1:1) through the gel purification column, collect a distilled liquid of 35~70mL, concentrate it to about 1mL in the rotary evaporator. Next, put it into a 5ml test tube, wash the rotatory evaporator with about 5ml of ethyl acetate several times and pour the washing liquid into the same tube. At the end, you can blow it to 1mL with nitrogen gas and prepare it ethyl acetate once again for future chromatographic analysis.

## A.5.3 Chromatogram condition

A.5.3.1 Chromatographic column: Elastic quartz capillary tube: 0.32mm in diameter and 30m long, painted with SE-54 and 0.25µm thick.

A.5.3.2 Column temperature: rising program:

60? /1 min 40? / min 110? 5? / min 235? 40? / min 265?

A.5.3.3 Entrance temperature: 270?

A.5.3.4 Checker: Flame -photometric detector (FPD-P), at a temperature of 270?

A.5.3.5 Gas: Nitrogen, blowing speed of 1ml/min, tail speed of 50ml/min.

A.5.3.6 Hydrogen and air flowing speeds: Hydrogen in 50ml/min and air in 500ml/min.

## A.5.4 Determination

Take the standard utilization liquid (A.2.9.2) of mixed organic phosphorous pesticides of 1µL and the purified solution of the sample (A.5.2) and put them into the chromatograph. Compare the peak height and area of the sample with that of the standard utilization liquid.

## A.5.5 Chromatogram of 13 organic phosphorous pesticides

For the chromatogram of 13 organic phosphorous pesticides, refer to Figure A.1.

- 1— ethamidophos,
- 2— dichlorvos,
- 3— acephate,
- 4— monocrotophos,
- 5— dimethoate,
- 6— disulfaton,
- 7— parathion-methyl,
- 8— fenitrothion,
- 9— pirimiphos methyl,
- 10— malathion,
- 11— fenthion,
- 12— parathion
- 13— ethion

(chart)

Figure A.1.

## A.6 How to describe the analysis result

Calculate the residue limit of a certain organic phosphorous left in the sample in Formula (A.1):

$$X = \frac{m_1 \times V_2 \times 1000}{m \times V_1 \times 1000} = \frac{m_1 \times V_2}{m \times V_1} \times 100 \dots \dots \dots (A.1)$$

Where:

X—The residue of a certain organic phosphorous pesticide left in the sample, in mg/kg,

m—The weight of a sample, in gram,



$m_1$ —The content of a certain organic phosphorous pesticide in the test liquid, in ng,

$V_1$ —The volume of a sample, in  $\mu\text{L}$ , and

$V_2$ —The final volume of the test liquid, in mL.

#### A.7 Allowable differences

The difference of a sample is not higher than the average value taken from the 2 determinations.

#### A.8 Accuracy

Take the recovery ratio as accurate

In light of demand, put the standard liquid of a certain organic phosphorous pesticide (A.2.9.2) into a piece of livestock or poultry meat, or an egg or milk and test at a recovery ratio of 70~110%.

You can compute the recovery ratio in Formula (A.2):

$$X = \frac{m_1 - m_2}{m} \times 100 \dots \dots \dots (A.2)$$

Where:

$Y$ —The recovery ratio, in %,

$m_1$ —The residue of a certain part found after a sample is added with standard liquid,

$m_2$ —The content of a certain part in a sample, and

$V_1$ —The quality of a certain part added.

END TRANSLATION